FACTORS AFFECTING PERSISTENCE OF DDT IN ARREDONDO FINE SAND

By

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DEDICATED TO THE MEMORY OF MY FATHER

THE LATE MOULVI ABDUR ROUF BHUIYA

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INTRODUCTION

Soil is rapidly being recognized as the principal reservoir of environmental pesticides. Because of its very nature, depth and high level of exposure it may also prove to be the most difficult to clean-up and methods for accelerating decay of these materials in soils would be of monumental importance whenever an undesirable accumulation occurs.

DDT[1] has been extensively used during the past 20 years in controlling various insect pests. As a result, contamination of the environment by the residual insecticide is almost universal. DDT and dieldrin are the most widespread insecticide residues in soils. On the average DDT persists the longest. This insecticide, present in agricultural soils, comes from foliage sprays or dusts which miss their targets and fall on the soil either close to the plants or after drifting, especially as very fine mists from low-volume sprays now commonly used. It has been estimated that probably as much as 50% of sprayed chemicals ultimately end up in soil either in this way or by being washed off the foliage. Large quantities of DDT to control soil-inhabiting pests are applied as sprays or dusts to the soil surface, and later cultivated into the soil, or are combined with fertilizer before it is distributed, or are coated on seeds before planting.

The mechanisms of disappearance of DDT from soil are still poorly understood, volatilization, codistillation, leaching, oxidation, hydrolysis, and microbial activity [illegible] in the process. Climate, soil physical and chemical conditions as determined by texture, aeration and drainage, moisture, organic matter, pH, mineral content, etc., may also affect the rate of breakdown.

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[1] See Table 1 for chemical designations of pesticides mentioned in text.

The persistent DDT residues contaminate crops grown in the soil. Hazards from such sources of contamination are magnified greatly if the chemical has been used repeatedly during growing seasons in the same locale.

According to the general trend in this field of research, it will be noted that interaction between pesticide chemicals and soil has been studied mostly under upland and not water-logged condition. Although such a trend is not curious from the type of crop cultivation in the world, another type of cultivation, i.e., paddy rice cultivation under water-logged condition, is most popular in Asia. In general, a drastic effect is induced on biochemical and chemical activity in the soil by submergence.

The breakdown of DDT may be affected by temperature. It is assumed that the insecticide decomposes far more rapidly during summer months when high soil temperature occurs, than during the winter.

From the above consideration, the objective of this laboratory study was to investigate the fate of applied technical-grade DDT in virgin Arredondo fine sand and the soil which had been altered with additions of activated sewage, sulfur, and calcium carbonate under well-aerated upland and water-logged conditions at 27C.

TABLE 1

COMMON AND CHEMICAL NAMES OF PESTICIDES MENTIONED IN TEXT

Aldrin	-	1,2,3,4,10,10-hexachloro-1,4,4a,5,8,8a-hexahydro-1,4-*endo*,*exo*-5,8-dimethanonaphthalene
BHC	-	Mixed isomers fo 1,2,3,4,5,6-hexachlorocyclohexane
Chlordane	-	1,2,4,5,6,7,10,10-octachloro-4,7,8,9,-tetrahydro-4,7-methyleneindane
p,p'-DDD(TDE)	-	1,1-dichloro-2,2-*bis*(*p*-chlorophenyl) ethane. The designation TDE is from tetrachlorodiphenylethane.
p,p'-DDE	-	1,1-dichloro-2,2-*bis*(*p*-chlorophenyl ethylene)
o,p'-DDT	-	1,1,1-trichloro-2-(*o*-chlorophenyl)-2-(*p*-chlorophenyl) ethane
p,p'-DDT	-	1,1,1-trichloro-2,2-*bis* (*p*-chlorophenyl ethane),
Dieldrin	-	1,2,3,4,10,10-hexachloro-6,7-epoxy-1,4,4a,5,6,7,8,8a-octahydro-1,4-*endo*,*exo*-5,8-dimethanonaphthalene.
Heptachlor	-	1,4,5,6,7,8,8-heptachloro-3a,4,5,5a-tetrahydro-4,7-*endo*-methanoindene.
Heptachlor epoxide	-	1,4,5,6,7,8,8-heptachloro-3a,2,3-epoxy-2,3,3a,7a-tetrahydro-4,7-methanoindene.
Lindane	-	Gamma isomer of 1,2,3,4,5,6-hexachlorocyclohexane
Toxaphene	-	Octochlorocamphene

REVIEW OF LITERATURE

Chemistry of DDT

The material now popularly known as DDT was first synthesized and described by Zeidler in 1874. The resynthesis of Zeidler's compound and its subsequent testing by the Swiss workers took place in 1939. The insecticidal properties of the compound were discovered in the Basle laboratories of the Swiss Company of J. R. Geigy S.A. by Dr. Paul Muller in the autumn of 1939 and patent application was made in Switzerland on March 7, 1940 (West and Campbell 1952).

The composition of the crude DDT prepared varies with the process employed and the conditions of reaction. Technical DDT is the form most widely used for the preparations of dusts and solutions.

Numerous gas chromatographic studies of technical-grade DDT have shown it to be composed of a number of compounds. It has been reported to be composed of at least 14 compounds, the principal ones being *p*,*p*'-DDT (65-80%), *o*,*p*'-DDT (11-30%), and *p*,*p*'-DDD (0.1-4.0%). p,p'-DDT is generally agreed to be the most active principle (Bliss and Gesten 1963).

Relative Stability of DDT as an Organochlorine Insecticide Residue in Soil

The chlorinated hydrocarbons have very stable structures and are lipophilic compounds of low polarity, but DDT, BHC, Chlordane and heptachlor are less stable in alkali than acid so pH may affect their persistence in soil (Edwards 1966). Some, such as BHC, break down to compounds that are not toxic to insects (Edwards et al. 1957, Lichten-

TABLE 2

APPROXIMATE COMPOSITION OF TECHNICAL DDT (FREAR 1955)

Compound	Approximate percentage
1,1,1-trichloro-2,2-*bis*(*p*-chlorophenyl) ethane (*p*,*p*'-DDT)	63-77
1,1,1-trichloro-2-(*o*-chlorophenyl-2-(*p*-chlorophenyl) ethane (*o*,*p*'-DDT)	8-21
1,1-dichloro-2,2-*bis*(*p*-chlorophenyl) ethane (*p*,*p*'-TDE)	0.3-4.0
1,1-dichloro-2-(*o*-chlorophenyl)-2-(*p*-chlorophenyl) ethane (*o*,*p*'-TDE)	0.04
1-*o*-chlorophenylethyl-2-trichloro-*p*-chlorobenzene sulfonate	0.1-1.9
2-trichloro-1-*p*-chlorophenylethanol	0.2
Bis(*p*-chlorophenyl) sulfone	0.03-0.6
a-Chloro-*a*-*p*-chlorophenylacetamide	0.01
a-Chloro-*a*-*o*-chlorophenylacetamide	0.01
Chlorobenzene	0.3
p-Dichlorobenzene	0.1
1,1,1,2-tetrachloro-2-(*p*-chlorophenyl) ethane	present
Sodium *p*-chlorobenzenesulfonate	0.02
Ammonium *p*-chlorobenzenesulfonate	0.01
Inorganic	0.01-0.1
Unidentified and losses	5.1-10.8

[illegible] and Schulz 1960). [illegible] yield a [illegible] of compounds, some of which are insecticidal and others not, an example is DDT, which breaks down in soil to DDD, DDE, and other compounds. Aldrin and heptachlor degrade to dieldrin and heptachlor epoxide which are insecticidally active (Edwards et al 1957, Bollen et al 1958, Gannon and Bigger 1958, Lichtenstein and Schulz 1960, Lichtenstein et al. 1964).

Allen et al (1954) treated soil with various insecticides and after 54 months, 39% of DDT and 6% of BHC remained in the soil.

Kincaid et al. (1960) found that 18% of DDT, 30% of DDD, and 24% of lindane disappeared in a year.

Lichtenstein et al (1960) treated several different field soils with chlorinated hydrocarbon insecticides and found that DDT was most persistent, aldrin least, and lindane intermediate. According to Roberts et al. (1962) DDT was the most persistent, about 36% remaining at the end of the 3rd year.

The variable persistence reported for DDT and other insecticides in soil reflects not only the effect of soil characteristics and environmental, climatic, or geographical variations, but different sampling methods and analytical techniques used by different workers.

DDT Concentration Affecting Persistence in the Soil

Kincaid et al (1960) found that the residues as percentage of the amount applied were much less for larger doses than small ones, e.g., after 88 months at 15 lb actual/acre 33% of DDT remained, but at 75 lb actual/acre only 19% remained.

Gollisher and Evans (1961) found no influence of concentration on the persistence of DDT in treated pasture soil in the range 1 to 8 lb actual/acre.

Lichtenstein and Schulz (1959) found that in the field, twice as much DDT as a percentage of the original application was recovered after 42 months from a plot treated with 100 lb actual/acre than from one treated with 10 lb actual/acre

Edwards (1968) plotted all the published results together with some unpublished data as regressions of percentage of original dose of insecticides against time The regressions showed clearly that proportionately more insecticides disappeared from a small dose than from a large one.

Soil Characteristics and DDT Persistence

Lambert et al (1965) list the following factors as being responsible for determining the fate of a chemical in the soil

A Type of soil:

1. Makeup, clay, silt, sand, organic matter, etc
2. Structure, bulk density, surface area, heterogeneity, etc
3. Prior treatment, chemical, agricultural practices, etc.

B Type of chemical: Physical properties, solubility, vapor pressure, stability, etc

C Climatic conditions: Rainfall, pressure, temperature, sunlight, etc

D Biological population: Types, nutrient requirements, adaptations, life cycles, etc

E Method of application: Granular, wettable powder, solvent, etc

The type of soil seems to be the most important single factor in determining the persistence and availability of the chemical. The soil type is determined by features such as the proportions of stones, sand silt, and clay, the mineral content and structure which influences the pH, and the organic matter content

Soil Type and Organic C[illegible]

Fleming and Maines (1953) studied the persistence of DDT in 24 mineral soils by bioassay with Japanese beetle larvae and found that DDT persisted longest in sand and least in muck. This is contrary to general belief that heavier soils with much clay and organic matter retain insecticides more strongly and that they persist there longer. But bioassay data estimates only that part of the insecticide which is available to kill the insect.

Appleman and Sears (1946) found that plants grown in soil treated with DDT were not so stunted as those grown in sand treated with DDT at the same rates. DDT up to 1,000 lb actual/acre can be more safely mixed (Edwards 1966) with muck soil than with mineral soils even when sensitive crops are grown.

Barlow and Hadaway (1955) showed that solid particles of DDT deposited from aqueous suspensions could disappear very rapidly from the external surfaces of blocks made from tropical soils. It was a physical process of adsorption and involved complete loss of biological activity of the insecticide. They found that the red soils with much iron oxide most actively sorbed DDT. British soils sorbed poorly, even peat sorbing much less than most of the African soils. Taveta red soils, which absorbed more slowly than the other red soils, contained a greater proportion of larger hard quartz-like fragments. They could even see under microscope the disappearance of DDT particles from surface of Uganda mud at about the same rate as from the surface of charcoal, clearly showing the considerable sorptive power of the lateritic soils.

Lichtenstein (1957), in a survey of 24 orchard and crop soils that had been treated with DDT between 1945 and 1955, found that the sandier soils contained the smallest percentage of the residues.

There is [illegible] [illegible] insecticide persistence, but a few investigations indicate that the larger clay contents increase the persistence ([illegible] 1966).

Lichtenstein and Schulz (1959) [illegible] that DDT, lindane, and aldrin persisted longer in a muck soil (organic matter content 40%) than in a silt loam (organic matter content 3.2%). Thirty-three percent of the originally applied DDT and lindane was recovered after 42 months from a muck soil and 4 to 7% of the originally applied aldrin, a Miami silt loam still contained 22.7% of the DDT, 3.5% of the lindane, and 1.1% of the aldrin after the same time. Soil type and organic matter content affected the persistence of DDT less than that of the other two chemicals.

Absorption by the inorganic and organic fractions of a soil may be attributed to one or more of the following forces:

1. Chemical adsorption which is due to coulombic forces.
2. Physical adsorption which can be attributed primarily to the interaction of dipoles (van der Waals forces).
3. Adsorption resulting from hydrogen bonding which is a bond between two highly electronegative atoms through the medium of the hydrogen atom.

Forces involved in physical adsorption are much weaker than those of hydrogen bonding, and these in turn are much weaker than those associated with chemical adsorption (Evans 1964). Due to the nonpolar nature of the DDT molecule, it is very unlikely that it is retained by the clay particle by any of the aforementioned adsorptive forces. The fact that DDT tends to associate or accumulate in the organic fraction may be due to compounds in the organic matter which act as solvents for the insecticide (Kunze 1966).

Soil pH

Chlorinated hydrocarbon insecticides are very stable and unreactive, and although DDT tends to be unstable in the presence of alkali, most of the evidence suggests that pH does not directly influence its persistence in the soil. Downs et al. (1951) gave data which indicated that alkalinity was not a significant factor in catalyzing DDT breakdown.

Fleming and Maines (1953) demonstrated that pH had no influence on the persistence of DDT in soil within the range of 4.0 to 7.5, and Smith (1948) concluded that DDT was stable in both acid and alkaline substrates.

Temperature, Rainfall, Soil Moisture, and Leaching

Gallaher and Evans (1961) considered that breakdown of DDT was not affected by temperature because decomposition rate did not increase over the summer period (25 to 35 weeks) with its high temperatures. If DDT is lost from soil by volatilization, chemical degradation or bacterial activity, all these processes could be affected by temperature. Solubility of the insecticide is also temperature-dependent and so this would also tend to decrease adsorption. As the temperature rises, more and more of the adsorbed insecticide becomes dissolved in soil moisture.

Barlow and Hadaway (1956) confirmed that deposits of DDT, BHC, and dieldrin on blocks of Uganda soil are sorbed at rates which decreased as humidity increased. They explained the results on the basis of competition between the water and the insecticides. Water appears to be adsorbed preferentially to the insecticides and increased amounts which are present in soils at high humidities have two effects. The

Initial disappearance of the insecticide [illegible] the [illegible] lying on the surface is retarded because water molecules already occupy a large proportion of the 'active' surface of the soil and can not be displaced. Once the insecticide is adsorbed, however, the same preferential adsorption of water results in an increased mobility of the insecticide molecules and therefore an enhanced rate of diffusion farther into and outside the soil.

The solubility of p, p'-DDT in water was found to be 1.2 ppb or less at 25C (Bowman et al 1960). Thus, there is unlikely to be much leaching as a direct result of solubility. It is possible, however, that the particles of the insecticide may be physically washed down through crevices into deeper soil. Guenzi and Beard (1967a) used columns of four soils to determine the amount of downward movement and persistence of DDT and lindane. DDT remained in the 0- to 3- cm layer in all soils regardless of the amount of water applied. Pesticide amended soils subjected to wetting and drying cycles lost more lindane than DDT. In the treatment receiving 35.4 cm of water during the cycling process, 57% of the applied lindane was recovered as compared to 78% of the DDT.

Allen et al (1954) applied DDT to soil, worked it into the soil to a depth of 6 inches, and 4 years later took cores of soil which they separated into 2-inch deep layers and analyzed them separately for residues. The top 6 inches of soil still contained 87% of the residues, the next 2 inches a further 9.5%, and the remaining 3.5% was in the 2 inches below this.

Because DDT is so extremely hydrophobic, it is involved in an interesting phenomenon. DDT molecules in water tend to migrate to the side of the container, where they may be adsorbed or adhere to the surface. At this water-air surface a process known as codistillation takes place (Acree et al 1963). The DDT is evaporated into the air along with water molecules at a much greater rate than would be predicted from its extremely low vapor pressure. Once the DDT is in the air, the catalytic action of sunlight promotes its destruction by oxidation.

Guenzi and Beard (1967b) using ^{14}C-labeled DDT in an incubation experiment in soil found no radioactivity in the hexane traps, indicating no volatilization of DDT.

Biodegradation, Occurrence, and Persistence of DDT and its Metabolites in Soil and Water Environments

Biodegradation

DDT residues in soils strongly resist microbial degradation, although dechlorination of DDT to DDD can occur in higher organisms (Datta et al 1964). In the rat, liver microsomal enzymes catalyze this reaction which also requires molecular oxygen and reduced triphosphopyridine nucleotide (Morello 1965). Also, dechlorination of DDT by microorganisms is possible. *Proteus vulgaris* and baker's yeast reductively dechlorinate DDT to DDD (Barker and Morrison 1965, Kallman and Andrews 1963). However, the efficacy of conversion by microorganisms is inversely proportional to availability of oxygen to the system. Stenersen (1965) found that when grown anaerobically or in an oxygen deficiency, the facultative anaerobes *Serratia marcescens*, *Escherichia coli* and an unidentified strain, converted DDT almost completely to DDD (about 90%). Castro (1964) and Miskus et al. (1965) found that dilute solutions of iron (II) porphyrin complexes were oxidized by DDT. Thus, it seemed likely that the microbial dechlorination of DDT might be explained as a process implicating the cytochromes of the respiratory chain, the absence of oxygen serving [illegible] DDT [illegible] [illegible] Aerobacter aerogenes, the use of selective inhibitors indicated that the reduced Fe (II) cytochrome oxidase was responsible for DDT dechlorination (Wedemeyer 1966) This finding may explain the persistence of DDT residues in soils and sediments

Chacko and Lockwood (1966) tested nine actinomycetes and eight fungi for their ability to degrade DDT The DDT was dechlorinated to DDD by six actinomycetes but not by any of the fungi This investigation was undertaken to discover microbes, particularly soil microbes that degrade DDT The rate of degradation of DDT by Streptomyces aureofaciens, S cinnamoneus, S viridochromogenes and Nocardia sp varied from 10 to 28%

Aerobacter aerogenes (ATC 13048) was shown to be capable of converting DDT to DDD by a pathway (Plimmer et al 1967) which appeared to be highly specific for DDT, since some closely related analogues did not behave similarly, ^{14}C-labeled DDT was used to follow the quantitative aspects of the reaction Mass spectrometry of the DDD which was produced demonstrated that a deuterium atom originally present at the 2 position of the DDT molecule was retained throughout the reaction It was concluded that the conversion of DDT to DDD by A aerogenes proceeds by reductive dechlorination and not by dehydrochlorination and subsequent reduction.

Feeding p,p'-DDT and o,p'-DDT separately (Klein et al 1964) at a level of 50 ppm in the diet of rats caused reductive dechlorination of DDT to DDD in the liver. No DDD was found in the fat DDE is not involved in the metabolic pathway Feeding o,p'-DDT yields evidence of isomeric conversion to p,p'-DDT in the fat stores

Guenzi and Beard (1967c) added DDT labeled with carbon-14 to soil [illegible] was incubated anaerobically in an atmosphere of 20% CO_2 and 80% N_2 at 30C for 2 weeks and 4 weeks. DDT and six possible decomposition products were separated by thin-layer chromatography, and the radioactivity of materials from individual spots were determined by liquid scintillation. The DDT was dechlorinated by soil microorganisms to DDD, and only traces of other degradation products were detected. No degradation was detected in sterile soil.

^{14}C-DDT and carrier DDT (Guenzi and Beard 1967b) were added to nonamended soil and 1% alfalfa-amended Pawnee soil. One set of soil samples, both sterile and nonsterile, was incubated anaerobically and another set of soil samples was incubated aerobically. DDT was converted to DDD in an anaerobic soil system by microorganisms and only traces of six other possible degradation products were detected. Amending the soil with 1% alfalfa increased the rate of conversion. After 6 months of aerobic incubation 75% of the added DDT was recovered in both the sterile and nonsterile soils. A maximum of 3% DDE and no DDD was detected during the incubation period. In addition, less than 1% of the carbon-14 in the phenyl ring of DDT was oxidized to CO_2 in the nonsterile soil.

Occurrence and Persistence in Soil and Water Environments

In the past, surveys to determine the levels of organochlorine residues in soil have been hampered by the lack of simple, yet accurate, analytical procedures. Colorimetric procedures, while adequate in some cases, were highly specific and time-consuming. Nevertheless, a number of surveys have been conducted with particular reference to DDT in orchard soils (Harris et al. 1966). The results of these studies have indicated that, generally, residues of DDT are higher in orchard soils and considerably lower in field crop soils. Harris et al. (1966) in exploratory) studies of the occurrence of organochlorine insecticide residues in agricultural soils in Southwestern Ontario found DDT in amounts exceeding 0.1 ppm in 24 of 32 soil samples, with highest being 118.9 ppm. DDE was present in all cases where DDT was detected and DDD was found in 13 of 26 samples containing DDT. Forty-five percent of all the soils investigated by Duffy and Wong (1967) in the Atlantic provinces of Canada contained residues of DDT plus metabolites between 1 and 5 ppm. A single Nova Scotia orchard sample contained 20 ppm and a single Newfoundland sample contained 17 ppm. The chief metabolites of DDT in the soils included DDE and DDD. The latter appeared chiefly in New Brunswick and Nova Scotia soil samples; DDE appeared in almost all soils containing DDT. The highest levels of DDE and DDD in the soils were 2.81 and 1.81 ppm respectively. *o*,*p*'-DDT was found consistently in DDT-treated soils. The highest concentration of *p*,*p*-DDT found in any soil sample was 2.63 ppm.

The subject of pesticides in natural water sources has been discussed by Hinden et al. (1964) as well as in the extensive review by Marth (1965). The former group reported tabulated data of pesticides found in water samples taken from many sources in the Columbia river basin in the irrigation seasons of 1961 and 1962. Demonstrable amounts of DDD were found in only six of the samples listed, but it is interesting to note that three of these contained higher concentrations of DDD than DDT, while in the other three samples the concentrations of DDD were only slightly less than those of DDT. DDD is often detected as a residue in situations where only DDT has been used, and DDD appears to persist for unusually long periods (Miscus et al. 1965). Studies with ^{14}C-DDT incubated with bovine rumen fluid, lake water, and aqueous solutions of reduced porphyrins showed partial conversion to ^{14}C-DDD

Conversion by bovine rumen fluid may explain certain DDD residues in milk, and conversion by lake water could account for the apparent extraordinary persistence of DDD in Clear Lake, California, because DDT may be available as a continuing precursor for DDD in these situations (Miscus et al. 1963). Distilled water, however, failed to show any conversion of DDT to DDD nor did boiled distilled water and rumen fluid. The fact that prophyries, under the proper reducing conditions, can convert DDT to DDD indicates one possible mechanism to explain this conversion in many biological systems.

MATERIALS AND METHODS

Collection and Preparation of the Soil and the Sampling of Sewage Used in the Experiment

Soil

Soil samples (0-15 cm) representing the Arredondo fine sand[1] soil type were obtained from the University of Florida Campus, immediately south of the McCarty Hall staff parking lot. The soil was air-dried at room temperature, sifted through a 2-mm screen and thoroughly mixed.

Sewage

The activated sewage was taken from the University of Florida Campus Sewage Treatment Plant.

Methods of analysis.--The soil pH was determined in 2:1, water to soil suspension after 30 minutes with a glass electrode and a Corning pH meter. The undiluted sewage was used for the pH determination.

Ten grams of air-dried soil were transferred to a weighed aluminum container with lid. The uncovered container was placed in an oven at 105C and dried for 12 hours for moisture determination (Piper 1947).

A raised border (a simple earth dike) was made around an area 8-ft x 8-ft, and sufficient water was added to wet the soil of the entire sampling area. The area was covered with a polyethylene sheet to prevent evaporation. The soil was allowed to drain for 2 days; the protective cover removed and soil samples taken. Five separate borings

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[1] United States Department of Agriculture. 1954. Soil survey of Alachua county, Florida. Series 1940, no. 10. 66 p.

[illegible] The water content was determined by the gravimetric method. The water percentage by weight at the time of sampling is defined as the field capacity (Peters 1965).

Organic carbon in the soil and the sewage was determined by the Walkley-Black wet-combustion method as modified by Walkley (1947). One gram of air-dried soil was oxidized with potassium dichromate solution and sulfuric acid. A 100-ml sample of sewage was measured out and poured into an Erlenmeyer flask, the solids being rinsed out of the measuring flask with distilled water and oxidized with potassium dichromate solution and sulfuric acid. With orthophenanthroline as an indicator, the excess dichromate was titrated against 1 N ferrous sulfate. Values for the organic carbon content have been reported as organic matter by multiplying the figure for organic carbon by the conventional "Van Bemmelen factor" of 1.724. The use of this figure is based on the assumption that organic matter contains 58% carbon.

The cation-exchange capacity was determined with ammonium following extraction of exchangeable cations. A 25-gram air-dried soil sample was weighed out into a 250-ml Erlenmeyer flask and 100 ml of neutral 1 N ammonium acetate was added. The flask was stoppered and shaken for several minutes and then allowed to stand overnight. The suspension was transferred to a Buchner funnel fitted with a moistened filter paper seated by gentle suction. The soil was leached with additional 400 ml of ammonium acetate. The electrolyte was washed out from the soil with 200 ml of 99% isopropyl alcohol. The soil was leached with 450 ml of 10% sodium chloride solution of pH 2.5 and the leachate collected in a suction flask. An aliquot was placed in an 800-ml Kjeldahl flask and the ammonium distilled and back titrated with 0.1 N sulfuric acid solution (Chapman 1965).

[illegible] solution[1] number 3 ([illegible]7 N ammonium acetate in 0.54 N acetic acid buffered at pH 4.8) was used for the extractable Ca, Mg, Na, and K determinations with the flame photometer.

Total N in the sewage was determined by the A.O.A.C. (1960) Kjeldahl method, modified by adding salicylic acid during the digestion to include nitrate-N in the determination. Ammonia was distilled into a 4% boric acid solution with a mixed indicator of bromocresol green and methyl red, and titrated with 0.1 N hydrochloric acid solution.

A 100-ml aliquot of sewage was pipetted into a weighed beaker and allowed to evaporate on a water bath. The dried material was placed in an oven at 70C for 4 hours, cooled and weighed.

Determination of sand, silt, and clay content in the soil was made by the pipette method (Day 1965).

The characteristics of the soil and sewage used in the experiment are summarized in Table 3.

Soil Treatments

Fig. 1 shows the outlines of the factorial experiment which was conducted in the completely randomized design.

Technical-grade DDT powder was supplied by Geigy Agricultural Chemicals. The insecticide, flowers of sulfur, and calcium carbonate powder were applied to 50-gram portions of air-dried soil and ground in a mortar with a pestle for thorough mixing. More air-dried soil was added to get the desired concentration, and the final mixing was done in a plastic rotary blender for 5 minutes. The sewage was applied and hand-mixed after the insecticide was added to the soil.

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[1] Southern Cooperative Series. 1965. Procedures used by state soil-testing laboratories in the southern region of the United States. Bull. 102. 49 p.

TABLE 1

CHARACTERISTICS OF THE SOIL AND THE SEWAGE USED IN THE EXPERIMENT

Soil	
Soil type	Arredondo fine sand
Depth sampled	0-15 cm
pH (H_2O)	5.65
Moisture in air-dried soil (oven-dry wt)	1.52%
Field capacity	15.28%
Organic carbon	1.71%
Organic matter	2.94%
Cation-exchange capacity	9.24 meq/100g
Extractable cations	
Ca (oven-dry wt)	810 ppm
Mg " " "	71 ppm
Na " " "	86 ppm
K " " "	22 ppm
Particle size distribution	
Sand (2.0-0.05 mm) (oven-dry wt)	93.5%
Silt (0.05-0.002 mm) " " "	3.0%
Clay (<0.002 mm) " " "	3.5%
Sewage	
pH	6.82
Organic carbon (w/v)	0.06%
Organic matter	0.10%
Dry matter	8.46%
Total N	0.04%

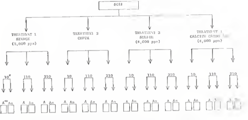

FIGURE 1 SCHEMATIC OUTLINES OF THE EXPERIMENT IN ARREDONDO FINE SAND

* Technical-grade DDT powder applied in ppm in the soil
**A Incubated at 37C at field capacity with water and replicated 2 [illegible]
An - Incubated at 37C by flooding with water and replicated 2 [illegible]

[illegible] grams of the treated soil were placed in a quart jar [illegible] with aluminum foil. The soil moisture was maintained at field capacity for aerobic conditions while anaerobic conditions were obtained by flooding with a constant head of 2 inches of distilled water over the soil surface. The experiment was conducted inside two incubators at 37C. The jars were weighed weekly and the moisture lost due to evaporation was replaced.

Key to Soil-Treatment Numbers

Incubated under field capacity conditions

$10A_1$ $10A_2$ Duplicate treatments with 10 ppm of technical-grade DDT

$110A_1$ $110A_2$ Duplicate treatments with 110 ppm of technical-grade DDT

$210A_1$ $210A_2$ Duplicate treatments with 210 ppm of technical-grade DDT

Incubated under anaerobic conditions in soil flooded with 2-inch head of water

$10An_1$ $10An_2$ Duplicate treatments with 10 ppm of technical-grade DDT

$110An_1$ $110An_2$ Duplicate treatments with 110 ppm of technical-grade DDT

$210An_1$ $210An_2$ Duplicate treatments with 210 ppm of technical-grade DDT

Soil Sampling

The soil-sampling schedule for the experiment was as follows:

Order of sampling	Weeks of incubation
1	0
2	6
3	12
4	18
5	24

[illegible] the contents in the jar were thoroughly mixed with a [illegible] Then a large plastic spoonful of soil was removed and stored in the freezer in a polyethylene bag

Analyses of DDT - Components and Metabolites

Gas Chromatography

Extraction of DDT[1] from the soil --The soil samples were air dried at room temperature. Large soil lumps were broken and the sample sifted through a 2-mm screen An air-dried soil sample, equivalent to 10-gram oven-dry wt, was placed in a quart jar and moistened with 1 5 ml of distilled water The extraction was accomplished with 100 ml of reagent grade hexane-acetone, 1:1 The jar was sealed and tumbled for 1 hour on an end-over-end tumbler at 64 rpm The extract was filtered through a fluted filter paper (R & D, no 515) into a 500-ml separatory funnel with teflon stopcock The extract was washed with three 100-ml portions of distilled water and the hexane layer retained The hexane layer was passed through a small Buchner funnel containing a filter paper (Whatman no 40) 30-ml anhydrous sodium sulfate, 15-ml super cel and 15-ml Celite 545 After clean-up the hexane layer was collected in a 50-ml glass-stoppered bottle and stored at 1 87C, prior to analysis.

Detection of residues --The gas chromatography equipment used consisted of a number of separate units designed to fit together to form a complete chromatography system. The chromatograph was equipped with electron-capture detector containing 200 mCi tritium foil and a strip chart recorder

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[1] Unpublished method Pesticide Research Laboratory, Food Science Department, IFAS, University of Florida Gainesville, Florida

[illegible] U-shaped 1 5-m [illegible] glass col[illegible] (6-[illegible] outside diameter) was packed with 3% QF-1 on 80/100 mesh Gas Chrom Q The operating parameters of the chromatograph were as follows

Carrier gas mixture	Argon 95% Methane 5%
Inlet pressure	40 psi
Flow rate	80 ml/min
Sensitivity	3×10^{-9} amp full scale
Chart speed	0 5 cm/min
Column temperature	180C
Detector temperature	202C
Vaporizer temperature	220C

The samples from a 10-microliter syringe of prepared standards and unknowns in hexane were used for analysis All samples were prepared by proper dilution in hexane so that the amount of pesticide residues and metabolites present would fall within the linear response-range of the detector

The retention times and responses established by injecting hexane solutions containing nanogram quantities of p,p'-DDT, p,p'-DDD, o,p'-DDT, and p,p'-DDE have been shown in Fig. 2, 3 and 4

Identification was based upon retention-time values, and the quantities of each material were calculated by relating the peak heights to those of standards analysed on the same day Responses obtained from the untreated soil extract and the reagent blank but not from the hexane solvent are shown in Table 4 and Figs 5 6, 7 and 8

A standard solution of the technical-grade DDT powder in hexane was chromatographed for the quantification of its components. The results are shown in Table 5 and Fig 9 and 10

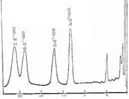

FIGURE 2 GAS CHROMATOGRAM OF 0.2 ng EACH OF *p*,*p*'-DDT *p*,*p*'-DDD, *o*,*p*'-DDT AND 0.1 ng OF *p*,*p*'-DDE STANDARDS IN HEXANE

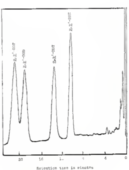

FIGURE 3 GAS CHROMATOGRAM OF 0.46 ng EACH OF p,p'-DDT, p,p'-DDD, o,p'-DDT, AND 0.23 ng OF p,p'-DDE STANDARDS IN HEXANE.

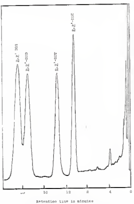

FIGURE 4 GAS CHROMATOGRAM OF 0.5 ng EACH OF *p,p'*-DDT, *p,p'*-DDD *o,p'*-DDT AND 0.3 ng OF *p,p'*-DDE STANDARDS IN HEXANE

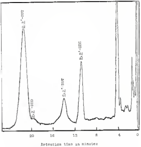

FIGURE 5 GAS CHROMATOGRAM OF DDT RESIDUES AND METABOLITES IN HEXANE EXTRACT FROM 0 - 14 cm DEPTH IN SOIL, PRIOR TO DDT TREATMENT

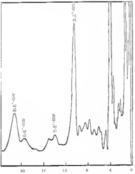

FIGURE 6: GAS CHROMATOGRAM OF DDT RESIDUES AND METABOLITES IN HEXANE EXTRACT FROM THE SOIL AT 14 - 30 cm DEPTH

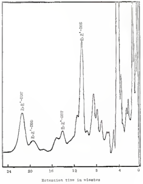

FIGURE 7 GAS CHROMATOGRAM OF DDT RESIDUES AND METABOLITES IN HEXANE EXTRACT AS REAGENT BLANK.

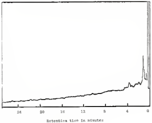

FIGURE 6 GAS CHROMATOGRAM OF HEXANE.

TABLE 4

DDT RESIDUES AND METABOLITES IN THE REAGENT BLANK AND IN THE SOIL PRIOR TO TREATMENT WITH TECHNICAL DDT

	ppm			
	p,p'-DDT	*p,p'*-DDD	*o,p'*-DDT	*p,p'*-DDE
Undiluted reagent blank	0.02	0.005	0.003	0.03
Soil 0- 14 cm depth, (oven-dry wt)	4.24	0.12	0.81	0.89
Soil 14- 30 cm depth (oven-dry wt)	-	-	-	0.12

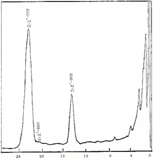

FIGURE 9: GAS CHROMATOGRAM OF 0.88 ng TECHNICAL DDT IN HEXANE.

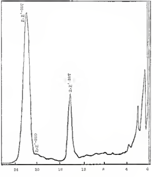

FIGURE 10 GAS CHROMATOGRAM OF 1.44 ng TECHNICAL DDT IN HEXANE

TABLE [illegible]

ANALYSIS OF TECHNICAL DDT

Replication number	Percent			
	p,p'-DDT	o,p'-DDT	p,p'-DDD	p,p'-DDE
1	57.63	26.38	0.17	-
2	63.12	28.64	0.21	-
Average	60.37	27.51	0.19	-

Colorimetric Determination of DDT (According to Martin and Batt 1958)

Method of extracting the soil.--The soil was prepared for analysis by drying in air and passing through a 2-mm screen. A representative 5-gram sample was then extracted for 3 hours in a micro-Soxhlett extraction apparatus using 60 ml of acetone.

Clean-up and the analytical procedures.--The acetone extract was evaporated to dryness in a stream of air at 35C. The residue was dissolved in 10 ml of carbon tetrachloride with warming and allowed to cool.

A glass tube, 200-mm x 16-mm was plugged with a small wad of cotton wool. It was packed with 10 grams of florisil on top of which 15 grams of alumina was packed. Three grams of anhydrous sodium sulfate were placed on top of the alumina layer. The extract contained in the 10 ml of carbon tetrachloride was carefully introduced into the column and as soon as the solvent had sunk into the alumina layer the column was washed with 4 x 10 ml of carbon tetrachloride. The solvent was evaporated to dryness in a stream of air.

Four ml of acetic acid were added and the beaker warmed to dissolve the residue, then 5 ml of 2-5% potassium permanganate solution were added. The beaker was placed on a boiling water bath for 10 minutes, allowed to cool, and decolorized by the addition, drop by drop, of a freshly prepared 5% sodium metabisulfite solution followed by 10 ml of water. Extraction was made successively, with vigorous shaking for 30 seconds each time, with 15-, 5- and 5- ml portions of carbon tetrachloride. The carbon tetrachloride extracts were combined and 10 ml of water added. A 4 N potassium hydroxide solution was added drop by drop with shaking until the aqueous layer was just alkaline to litmus paper. The carbon tetrachloride layer was drained off and the aqueous layer washed with a little carbon tetrachloride. The combined carbon tetrachloride layers were washed with 5 ml of water.

The carbon tetrachloride extract was run through anhydrous sodium sulfate to remove water. The desiccant was washed with a little more solvent and the extract evaporated to dryness.

Two ml of nitration acid (fuming nitric acid and concentrated sulfuric acid, 1:1) were added and the beaker with a cover glass was placed in a boiling water bath for 10 minutes. After cooling, 10 ml of water were slowly added. The contents in the beaker were cooled again and neutralized by the addition, with swirling and cooling, of 4 *N* potassium hydroxide solution until litmus paper just turned blue. Then 2 drops of 4 *N* potassium hydroxide solution were added in excess.

The cooled solution was washed into a 60- ml separatory funnel with 5 ml of water. The beaker was rinsed with 15 ml of carbon tetrachloride and the washing added to the funnel and extracted, after vigorous shaking for 30 seconds. This was reextracted with 2 additional 5- ml portions of carbon tetrachloride, rinsing the beaker each time. The carbon tetrachloride extracts were combined and washed twice with 5- ml portions of water.

The carbon tetrachloride solution was run through anhydrous sodium sulfate. The funnel and the desiccant were washed with a little more solvent. The volume was reduced to 0.5 ml in a current of air.

Ten ml of 5% ethanolic potassium hydroxide solution were added. The ethanolic potassium hydroxide solution was prepared by heating 10 grams of potassium hydroxide with 100 ml of absolute ethanol under reflux until dissolution was complete. The solution was freshly prepared and filtered immediately before use. The contents were shaken gently for 15 minutes, and OD measured at 600 millimicrons wavelength.

RESULTS AND DISCUSSION

The Treatment Effect on pH

The statistical analysis of the data is given in Appendix Table 26.

As shown in Appendix Tables 28-47 and Fig. 11-14, there was a response in pH with the addition of sulfur and calcium carbonate applied at the rate of 4,000 ppm.

Flowers of sulfur (Appendix Tables 38-42, and Fig. 13) reacted rather quickly in lowering the pH from about 8 to 3 during the 8- week period of incubation. Incubation at 37C and moisture at field capacity responded early, whereas flooded soils showed similar pH values after 12 weeks. Because the degree of aeration is inversely related to soil moisture, it is to be expected that at high moisture content oxygen will be a limiting factor, and because the sulfur oxidizing organisms are aerobic, the decrease in the rate of oxidation at high moisture levels undoubtedly was related to the decreased supply of oxygen. The finer the particle size of a given mass of sulfur, the larger the specific surface and the faster the conversion to sulfate.

The calcium carbonate treatment exhibited an increase in pH and then decreased with time (Appendix Tables 43-47, and Fig. 14).

The sewage treatment did not produce any effect on pH change (Appendix Tables 28-32, and Fig. 11).

In general, flooded soils had approximately one unit higher pH than the soil kept at field capacity. It is known that after submer-

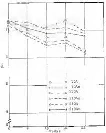

FIGURE 11. pH CHANGES IN TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT AND ACTIVATED SEWAGE DURING 24 WEEKS.

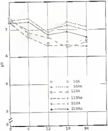

FIGURE 12. pH CHANGES IN TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT DURING 24 WEEKS.

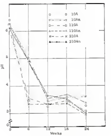

FIGURE 13 pH CHANGES IN TREATMENT 3 CONTAINING SOIL, TECHNICAL DDT AND SULFUR DURING 24 WEEKS,

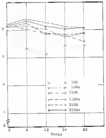

FIGURE 14. pH CHANGES IN TREATMENT 4 CONTAINING SOIL, TECHNICAL DDT AND CALCIUM CARBONATE DURING 24 WEEKS.

[illegible] using [illegible].

[illegible] hydrocarbon [illegible] Although DDT tends to be [illegible] presence of [illegible] evidence suggests that [illegible] directly influence [illegible] in soil. Downs et al ([illegible]) presented data which indicated [illegible] was not a significant factor in catalysing DDT breakdown.

The pH range for acid soils ordinarily [illegible] around 4 to 7. Values much below 4 are obtained only when [illegible] acids are present, such as H_2SO_4 in "cat clays". Values above 7 indicate alkalinity rather than acidity.

The purpose of sulfur and calcium carbonate treatments was to get extreme pH values in the substrates and to observe the fate of technical-grade DDT under the conditions of the experiment. Appendix Tables 28-47, and Fig. 11, 12, 13, and 14 show that during 21 weeks of incubation, pH values of the four treatment combinations ranged from about 3 to 7.

Degradation of Technical DDT Components and the Detection of Metabolites

Persistence of DDT in soils is well-documented (Edwards 1966), and methods for accelerating the decay of this material in soils would be of monumental importance whenever an undesirable accumulation occurs.

Guenzi and Beard (1967c) added ^{14}C-labeled p,p'-DDT to soil and the mixture was incubated anaerobically for 2 weeks and 4 weeks. p,p'-DDT and six possible decomposition products were separated by thin-layer chromatography and the radioactivity of the material from individual spots was determined by liquid scintillation. The *p,p'*-DDT was dechlorinated to *p,p'*-DDD and only traces of *p,p'*-DDA, *p,p'*-DDE, and other metabolites were detected.

Technical-grade DDT is composed of at least 14 compounds, the principal ones being *p,p'*-DDT, *o,p'*-DDT, and *p,p'*-DDD (Blinn and Gaston 1963). The insecticide used in this experiment was analyzed for *p,p'*-DDT, *o,p'*-DDT, and *p,p'*-DDD and the results are shown in Table 5.

In view of the above considerations, the operating parameters of the gas chromatograph were adjusted for the detection and quantification of *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE as the technical-grade DDT-components and metabolites.

The results of the gas chromatographic analyses are given in Tables 6-25. The analysis of variance data for *p,p'*-DDT, *p,p'*-DDD, *o,p'*-DDT, and *p,p'*-DDE are presented in Appendix Table 27.

Three levels of applied technical-grade DDT showed significant differences on the levels of DDT-components and metabolites and the response was linear. This was because different amounts of *p,p'*-DDT, *p,p'*-DDD, and *o,p'*-DDT were present in the technical-grade DDT which was applied at 10 ppm, 110 ppm, and 210 ppm levels.

The analysis of variance data for the mean squares for DDT-components and metabolites showed a positive response due to incubation at 37C at field capacity versus flooding with water. The mean squares increased significantly with time. Data from the last sampling after 24 weeks of incubation showed a curvilinear trend which can be compared with the typical growth curve obtained in biological responses.

Considering the amounts of *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE as the main effect or as the source of variation in their analyses, significant differences were obtained. This shows that measurements made for the components were significantly different. This is because of sampling and experimental error associated with the heterogeneity of the soil. Because of the plastic nature of the technical-grade DDT powder, it has the tendency to stick to solid surfaces, and the small amounts of the insecticide could not be mixed thoroughly with the soil mass.

The mean squares of the data also show that the totals for *p,p'*-DDT, *p,p'*-DDD, *o,p'*-DDT, and *p,p'*-DDE decreased gradually with the increase in incubation time. Guenzi and Beard (1967c) separated seven possible decomposition products due to anaerobic biodegradation of *p,p'*-DDT alone. It seems likely that other compounds are also formed by the degradation of technical-grade DDT-components of which only four were accounted for in this study.

The interactions between four treatments and the DDT- components and metabolites were not significantly different. Or, in other words, four treatments consisting of sewage, no treatment, sulfur, and calcium carbonate did not have any significant interaction with the levels of *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE as degradation products. It is also apparent that pH changes did not affect the breakdown of DDT.

It is to be expected that the interaction between the three levels of technical-grade DDT and the components analyzed for, should be significant and is due to different amounts of *p,p'*-DDT, *o,p'*-DDT, and *p,p'*-DDD present in the three levels of technical-grade DDT applied.

The interaction among four treatment combinations, levels of technical-grade DDT applied, and DDT-components and metabolites was not significant.

The interaction between the components analyzed and incubation at

37C at field capacity versus flooding with water became significant after the 6-week period and throughout until the end of the experiment. The mean squares in the analysis of variance data increased after 6- and 12- week periods.

The four treatment combinations did not have any appreciable effect on the degradation of the insecticide. It was, therefore, decided to consider the average of *p,p*'-DDT, *p,p*'-DDD, *o,p*'-DDT, and *p,p*'-DDE separately from the four treatment combinations. Bar charts were drawn for the three levels of applied technical-grade DDT.

Bar charts for each level of technical-grade DDT applied at the rate of 10 ppm, 110 ppm, and 210 ppm are shown in Figs. 15-20. One set of three bar charts, shown in Figs. 15, 17, and 19, represents the change in concentration in ppm of *p,p*'-DDT, *p,p*'-DDD, *o,p*'-DDT, and *p,p*'-DDE as a function of incubation time at field capacity. Another set, Figs. 16, 18, and 20, represents the water-logged soil.

The average of *p,p*'-DDT, *p,p*'-DDD, *o,p*'-DDT, and *p,p*'-DDE concentrations in ppm detected and measured from 110 ppm technical-grade DDT treatments are given as bar charts in Figs. 21, 22, 23, and 24. The change in concentration of each of the four compounds detected in the soil maintained at field capacity and under water has been plotted side by side, as a function of time, for easy comparison. The 110 ppm technical-grade DDT treatment was chosen to show the comparison as 10 ppm and 210 ppm treatments show similar trends. Also, 110 ppm level is the intermediate between the two extreme concentrations applied.

p,p'-DDT and *p,p*'-DDD

Because the standard Schechter-Haller method for colorimetric analysis of DDT does not differentiate *p,p*'-DDT from *p,p*'-DDD, a principal metabolite of the former, it is not surprising that the conversion to

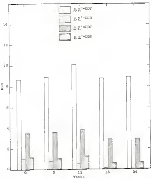

FIGURE 15 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *p*,*p*'-DDT, *p*,*p*'-DDD, *o*,*p*'-DDT, AND *p*,*p*'-DDE IN 10 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AS A FUNCTION OF INCUBATION TIME.

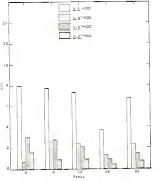

FIGURE 16 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF p,p'-DDT p,p'-DDD o,p'-DDT, AND p,p'-DDE IN 10 ppm TECHNICAL DDT TREATMENTS UNDER WATER AS A FUNCTION OF INCUBATION TIME.

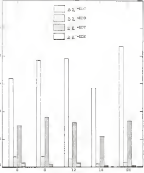

FIGURE 17. BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF p,p'-DDT, p,p'-DDD, o,p'-DDT, AND p,p'-DDE IN 110 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AS A FUNCTION OF INCUBATION TIME.

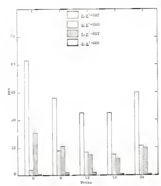

FIGURE 18. BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *p*,*p*'-DDT, *p*,*p*'-DDD, *o*,*p*'-DDT, AND *p*,*p*'-DDE IN 110 ppm TECHNICAL DDT TREATMENTS UNDER WATER AS A FUNCTION OF INCUBATION TIME.

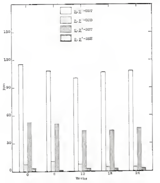

FIGURE 19 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *p,p'*-DDT, *p,p'*-DDD, *o,p'*-DDT, AND *p,p'*-DDE IN 210 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AS A FUNCTION OF INCUBATION TIME

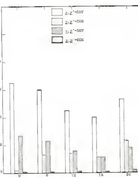

FIGURE 20 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF p,p -DDT p,p -DDD o,p -DDT AND p,p'-DDE IN 210 ppm TECHNICAL DDT TREATMENTS UNDER WATER AS A FUNCTION OF INCUBATION TIME

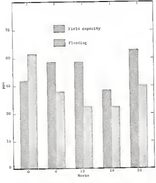

FIGURE 21. BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *p p'*-DDT IN 110 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AND UNDER WATER AS A FUNCTION OF INCUBATION TIME.

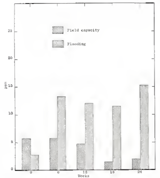

FIGURE 22 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *p,p* -DDD IN 110 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AND UNDER WATER AS A FUNCTION OF INCUBATION TIME

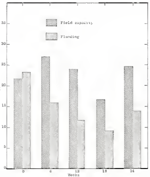

FIGURE 23 BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF *o,p* -DDT IN 110 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AND UNDER WATER AS A FUNCTION OF INCUBATION TIME.

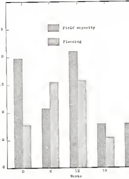

FIGURE 24. BAR CHART SHOWING THE CHANGE IN CONCENTRATION OF p,p -DDE IN 110 ppm TECHNICAL DDT TREATMENTS AT FIELD CAPACITY AND UNDER WATER AS A FUNCTION OF INCUBATION TIME

p,p'-DDD was not observed (Miskus et al. 1965). The results from colorimetric analyses for DDT are shown in Appendix Tables 28-47.

Electron-capture gas chromatography now provides a means of studying DDT and its degradation products with speed, sensitivity, and precision not believed possible a few years ago. This investigation employed that technique, which was designed to measure *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE.

The data on *p,p'*-DDT, *o,p'*-DDT, *p,p'*-DDD, and *p,p'*-DDE are entered in Tables 8-25. The responses obtained as gas chromatograms are presented in Fig. 25-30.

10 ppm technical-grade DDT treatments.--*p,p'*-DDT was dechlorinated to *p,p'*-DDD in the flooded soil. Analysis of the soil samples at the end of 0, 6, 12, 18, and 24 weeks of incubation showed that in the flooded soil *p,p'*-DDT content decreased from around 8 ppm to 3 ppm. *p,p'*-DDD concentration rose from around 0.6 ppm to 2.8 ppm (Fig. 15, 16, 25, and 26). The level of *p,p'*-DDD declined from 1 ppm to 0.14 ppm in the soil kept at field capacity.

The work of Miskus et al. (1965), in which *p,p'*-DDT was found to be converted to *p,p'*-DDD in samples of lake water and in stagnating rumen fluid,suggests that conversion is enhanced under conditions favoring anaerobiosis. Direct evidence for the potentiation of dechlorination of *p,p'*-DDT by microorganisms under anaerobic conditions has been presented by Stenersen (1965), and Wedemeyer (1968). Each noted that the efficacy of degradation of DDT by bacteria is inversely proportional to the availability of oxygen in the environment. Guenzi and Beard (1967c) added ^{14}C-labeled *p,p'*-DDT to soil, and the mixture was incubated anaerobically in an atmosphere of 20% CO_2 and 80% N_2. The soil moisture was adjusted to 28%. The *p,p'*-DDT was dechlorinated by soil microorganisms to *p,p'*-DDD. No degradation was detected in the sterile soil.

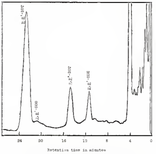

FIGURE 25 GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 2 CONTAINING SOIL AND 10 ppm TECHNICAL DDT, INCUBATED AT 37C FOR 12 WEEKS AT FIELD CAPACITY.

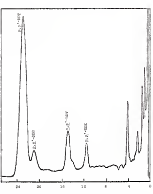

FIGURE 26 GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 1 CONTAINING SOIL, 10 ppm TECHNICAL DDT, AND ACTIVATED SEWAGE, INCUBATED AT 37C FOR 12 WEEKS UNDER WATER.

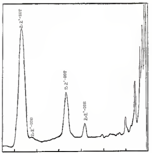

FIGURE 27 GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 1 CONTAINING SOIL, 110 ppm TECHNICAL DDT, AND ACTIVATED SEWAGE INCUBATED AT 37C FOR 12 WEEKS AT FIELD CAPACITY

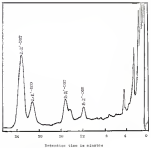

FIGURE 28. GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 1 CONTAINING SOIL, 110 ppm TECHNICAL DDT AND ACTIVATED SEWAGE INCUBATED AT 37C FOR 12 WEEKS UNDER WATER.

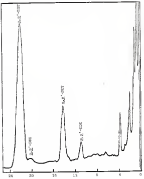

FIGURE 29. GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 4 CONTAINING SOIL, 210 ppm TECHNICAL DDT, AND CALCIUM CARBONATE, INCUBATED AT 37C FOR 12 WEEKS AT FIELD CAPACITY.

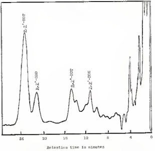

FIGURE 30 GAS CHROMATOGRAM OF DDT - COMPONENTS AND METABOLITES IN HEXANE EXTRACT FROM TREATMENT 4 CONTAINING SOIL, 210 ppm TECHNICAL DDT, AND CALCIUM CARBONATE, INCUBATED AT 37C FOR 12 WEEKS UNDER WATER.

Because the degree of aeration is inversely related to soil moisture, it is to be expected that under flooding with water, oxygen will be a limiting factor. The observed dechlorination of *p,p'*-DDT and its conversion to *p,p'*-DDD was enhanced under water-logged conditions creating or favoring anaerobiosis (Fig. 15, 16, 23, and 26).

110 ppm technical-grade DDT treatments.--Anaerobic conditions obtained by flooding caused reductive dechlorination of *p,p'*-DDT to the extent that a final concentration of 15.4 ppm of *p,p'*-DDD was recorded. In the soil maintained at field capacity the *p,p'*-DDD concentration decreased from 5.7 ppm to 1.5 ppm at the end of incubation. The concentration of *p,p'*-DDT decreased from around 82 ppm to 33 ppm in the flooded soil only and no noticeable change occurred in the well-drained situation (Fig. 17, 18, 21, 22, 27 and 28).

210 ppm technical-grade DDT treatments.--At the end of incubation, 35 ppm of *p,p'*-DDD was detected in the soil kept under water. Analysis of the moist soil showed an increase to 10 ppm of *p,p'*-DDD presumably due to localized anaerobiosis caused by entrapped CO_2 or water. However, with time, *p,p'*-DDD degraded and a lower level of 3 ppm was detected. *p,p'*-DDT showed recalcitrance in the moist soil but in the flooded soil it decreased from 98 ppm to 50 ppm (Fig. 19, 20, 29, and 30).

o,p'-DDT

o,p'-DDT is the major constituent of technical-grade DDT (27.5%), and as a biocide much less toxic than *p,p'*-DDT.

Studies on the degradation and occurrence of *o,p'*-DDT in soil are few. Duffy and Wong (1967), in their investigations on the occurrence of organochlorine insecticide residues, detected *o,p'*-DDD consistently in DDT- treated soils. The highest concentration of *o,p'*-DDT found in any soil sample was 2.63 ppm.

Kleis et al (1964) found that when rats were fed o,p'-DDT there was clear evidence of a biological isomeric transformation of o,p'-DDT to p,p'-DDT in the fat stores.

10 ppm technical-grade DDT treatments.--o,p'-DDT breakdown did occur in the soil maintained at field capacity and in water-logged state. The loss was more significant from the flooded soil where from a 3 ppm concentration at 0 time, 0.8 ppm was reached after 24 weeks of incubation (Fig. 15, 16, 25, and 26). As found by Kleis et al. (1964), experimenting with rats, in both situations in the soil isomeric transformation of o,p'-DDT to p,p'-DDT could take place.

110 ppm technical-grade DDT treatments.--From a concentration around 23 ppm, o,p'-DDT degraded to 9 ppm under anaerobic conditions. The compound practically remained unaltered in the aerobic soil (Fig. 17, 18, 23, 27, and 28).

210 ppm technical-grade DDT treatments.--Some alteration of o,p'-DDT was noticeable in the moist soil and from an initial concentration of 53 ppm it decreased to 43 ppm (Fig. 18, 20, 29, and 30). More o,p'-DDT was lost from the flooded soil where from an initial concentration of 40 ppm at the initiation of incubation only 17 ppm was detected at the end of 24 weeks.

p,p'-DDE

Duffy and Wong (1967) observed that p,p'-DDD and p,p'-DDE are the chief metabolites of DDT in the soil. p,p'-DDE occurred in all soils containing DDT. Guenzi and Beard (1967c) isolated traces of p,p'-DDE as one of the seven metabolites of p,p'-DDT after anaerobic incubation of soil containing p,p'-DDT.

10 ppm, 110 ppm, and 210 ppm technical-grade DDT treatments.--This study found similar amounts of p,p'-DDE in all of the three levels of technical-grade DDT treatments. The concentrations ranging from 0.4 ppm to 2.6 ppm were not significantly different under both aerobic and anaerobic conditions (Fig. 15-20, 24, and 25-30).

Effect of Temperature on the Degradation of the Insecticide

It is the general belief that the decomposition of the insecticide is greatly effected by temperature. However, Gallaher and Evans (1961) found that the breakdown of DDT was not affected by temperature because the decomposition rate did not increase over the summer period (25 to 35 weeks) with its accompanying higher temperatures.

Temperature is one of the primary environmental variables influencing soil bacteria. Most microorganisms are mesophiles with optima in the vicinity of 25 to 35C. Mesophilic types constitute the bulk of the soil bacteria.

Guenzi and Beard (1967a) observed anaerobic biodegradation of *p,p'*-DDT in an atmosphere of 20% CO_2 and 80% N_2 in the soil which was incubated for 2- and 4- week periods at 30C.

This study was performed at an incubation temperature of 37C and degradation of *p,p'*-DDT was observed mostly in the anaerobic situation under water. Since temperature was not a variable in this investigation it may be safe to conclude that other than the indirect effect on soil microbes, it was not directly involved in the degradation of the insecticide through the codistillation process as mentioned by Acree et al. (1963).

TABLE 6

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT, AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	9.58	0.93	3.64	1.50
$10A_2$	7.28	0.77	3.48	1.16
$110A_1$	57.78	4.85	25.24	1.64
$110A_2$	39.54	2.88	17.15	0.65
$210A_1$	61.02	8.48	38.87	1.69
$210A_2$	103.24	7.30	47.81	2.74
$10An_1$	10.78	1.07	5.29	1.22
$10An_2$	6.68	0.45	2.51	1.21
$110An_1$	60.16	1.38	20.41	0.42
$110An_2$	73.76	2.72	28.78	0.83
$210An_1$	78.84	1.72	34.52	0.33
$210An_2$	60.36	2.00	22.67	0.37

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* 1st sampling after 0 week of incubation.

** As ppm oven-dry soil.

***Technical-grade DDT in ppm applied in the soil and replicated 2 times.

TABLE 7

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
10A$_1$***	6.36	0.15	2.68	1.02
10A$_2$	9.57	0.57	3.68	1.23
110A$_1$	54.04	5.75	28.57	1.33
110A$_2$	58.33	7.36	29.60	1.74
210A$_1$	104.02	5.20	44.38	0.14
210A$_2$	126.10	4.56	52.99	0.14
10An$_1$	8.34	1.47	1.30	0.65
10An$_2$	15.45	2.20	4.41	0.76
110An$_1$	52.79	16.06	22.03	1.37
110An$_2$	53.77	18.70	19.63	1.87
210An$_1$	78.34	12.72	26.18	0.12
210An$_2$	67.06	18.24	21.33	0.21

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* 2nd sampling after 8 weeks of incubation

** As ppm oven-dry soil.

***Technical-grade DDT in ppm applied in the soil and replicated 2 times

TABLE

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT, AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	10.57	0.69	3.64	1.30
$10A_2$	12.52	0.99	4.74	1.59
$110A_1$	61.47	4.39	24.70	2.19
$110A_2$	55.14	4.12	23.19	2.06
$210A_1$	104.80	9.26	42.17	3.59
$210A_2$	92.91	4.93	35.47	3.44
$10An_1$	6.78	2.98	2.52	0.89
$10An_2$	4.77	3.19	1.41	0.88
$110An_1$	30.51	13.15	11.57	1.59
$110An_2$	31.92	13.81	11.91	1.43
$210An_1$	40.94	18.61	15.92	1.30
$210An_2$	58.84	18.31	22.88	1.56

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[*] 3rd sampling after 12 weeks of incubation.

[**] As ppm oven-dry soil

[***] Technical-grade DDT in ppm applied in the soil and replicated 2 times.

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS
AND METABOLITES FROM TREATMENT 1 CONTAINING
SOIL TECHNICAL DDT, AND ACTIVATED SEWAGE,
INCUBATED AT 37C AT FIELD CAPACITY AND BY
FLOODING WITH WATER *

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	11.53	0.22	3.42	0.72
$10A_2$	8.38	0.30	2.88	0.80
$110A_1$	44.08	1.97	15.85	0.48
$110A_2$	44.18	0.78	18.30	0.74
$210A_1$	118.95	4.10	44.08	1.64
$210A_2$	104.28	3.78	41.71	1.33
$10An_1$	5.88	1.28	1.73	0.31
$10An_2$	3.46	1.30	0.34	0.34
$110An_1$	47.03	21.78	13.36	0.78
$110An_2$	45.40	22.03	13.31	0.92
$210An_1$	74.37	23.11	25.12	1.12
$210An_2$	76.42	27.21	26.17	1.87

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT in ppm applied in the soil and replicated 2 times

TABLE 10

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT COMPONENTS AND METABOLITES FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	12.27	0.35	6.70	0.67
$10A_2$	8.75	0.28	3.27	0.65
$110A_1$	78.56	0.17	23.50	0.12
$110A_2$	68.38	0.23	24.31	0.76
$210A_1$	110.55	4.02	48.23	1.40
$210A_2$	84.42	5.00	46.23	1.40
$10An_1$	6.07	1.83	1.44	0.55
$10An_2$	6.37	1.66	1.69	0.51
$110An_1$	38.16	8.91	7.62	0.11
$110An_2$	22.54	8.76	1.35	0.06
$210An_1$	54.11	20.29	19.36	0.86
$210An_2$	99.56	29.77	34.43	1.30

* 5th sampling after 24 weeks of incubation.

** As ppm oven-dry soil

*** Technical-grade DDT in ppm applied in the soil and replicated 2 times

TABLE 11

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT - components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	8.65	0.88	3.42	1.26
$10A_2$	7.33	0.80	3.35	1.05
$110A_1$	49.16	4.20	19.19	0.33
$110A_2$	52.15	3.74	25.25	0.55
$210A_1$	149.78	7.88	66.01	1.97
$210A_2$	104.69	4.18	47.11	1.87
$10An_1$	8.35	0.37	3.45	1.88
$10An_2$	7.25	0.54	2.64	1.79
$110An_1$	55.91	2.80	16.93	1.01
$110An_2$	53.75	1.46	17.10	1.07
$210An_1$	97.22	2.62	43.47	0.62
$210An_2$	89.55	2.15	34.09	0.72

* 1st sampling after 0 week of incubation

** As ppm oven-dry soil

***Technical-grade DDT in ppm applied in the soil and replicated 2 times.

TABLE 12

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	DDT - components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	8.27	0.08	2.85	0.88
$10A_2$	8.83	0.08	3.11	0.89
$110A_1$	58.45	8.83	29.93	1.58
$110A_2$	59.88	8.04	29.02	1.78
$210A_1$	104.78	5.78	50.58	0.36
$210A_2$	100.00	5.15	51.47	0.28
$10An_1$	8.45	1.88	1.78	0.65
$10An_2$	7.12	2.86	2.32	1.08
$110An_1$	43.22	18.88	18.81	3.81
$110An_2$	47.22	17.78	20.10	4.20
$210An_1$	98.25	13.19	34.82	2.84
$210An_2$	102.30	18.47	39.07	2.48

* 2nd sampling after 8 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT in ppm applied in the soil and replicated 2 times

TABLE 13

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT - components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	12.08	0.50	4.75	1.58
$10A_2$	12.31	0.34	5.43	1.69
$110A_1$	52.55	3.60	23.15	1.54
$110A_2$	62.44	3.21	26.63	2.29
$210A_1$	83.83	11.36	37.65	1.85
$210A_2$	104.78	5.25	43.28	2.82
$10An_1$	13.24	2.25	3.33	0.72
$10An_2$	12.98	2.20	2.82	0.82
$110An_1$	26.80	11.53	8.78	1.51
$110An_2$	30.33	11.68	8.88	2.33
$210An_1$	80.21	23.13	25.12	1.54
$210An_2$	84.02	27.18	32.12	1.15

* 3rd sampling after 12 weeks of incubation.

** As ppm oven-dry soil.

*** Technical-grade DDT in ppm applied in the soil and replicated 2 times

TABLE 14

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	DDT - components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	8.20	0.15	2.80	0.35
$10A_2$	8.47	0.28	2.68	0.96
$110A_1$	32.67	0.65	11.91	0.66
$110A_2$	45.62	0.70	17.76	0.93
$210A_1$	63.01	5.34	37.41	1.48
$210A_2$	111.66	6.51	45.59	1.48
$10An_1$	2.97	1.53	0.76	0.46
$10An_2$	3.16	1.63	0.81	0.47
$110An_1$	35.67	6.23	11.52	0.38
$110An_2$	17.62	7.14	3.81	0.66
$210An_1$	74.33	15.70	16.84	0.94
$210An_2$	56.64	14.24	15.60	0.65

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* 4th sampling after 16 weeks of incubation.

** As ppm oven-dry soil.

*** Technical-grade DDT in ppm applied to the soil and replicated 2 times.

TABLE 15

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER*

Treatment number	DDT - components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	8.23	0.06	1.32	0.25
$10A_2$	8.28	0.05	1.97	0.31
$110A_1$	81.71	0.27	30.29	0.73
$110A_2$	93.81	0.23	28.08	0.56
$210A_1$	130.65	8.03	48.22	1.50
$210A_2$	109.80	4.85	44.88	1.87
$10An_1$	5.92	1.23	0.74	0.40
$10An_2$	3.00	0.87	0.35	0.21
$110An_1$	85.65	23.23	31.81	0.33
$110An_2$	26.28	4.54	3.03	0.75
$210An_1$	42.21	19.09	12.06	0.80
$210An_2$	54.18	18.70	17.73	0.78

* 5th sampling after 24 weeks of incubation

** As ppm oven-dry soil.

*** Technical-grade DDT in ppm applied in the soil and replicated 2 times.

TABLE 18

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 3 CONTAINING SOIL, TECHNICAL DDT, AND SULFUR, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	*p,p'*-DDD	*o,p'*-DDT	*p,p'*-DDE
$10A_1$***	8.05	1.09	3.60	1.20
$10A_2$	7.81	0.93	2.30	1.17
$110A_1$	43.51	8.16	21.48	2.69
$110A_2$	42.58	7.62	20.56	3.33
$210A_1$	121.44	6.28	53.30	3.14
$210A_2$	120.60	8.04	51.50	3.05
$10An_1$	9.20	0.92	2.73	1.54
$10An_2$	8.82	0.99	2.80	1.63
$110An_1$	58.69	2.95	26.20	0.66
$110An_2$	61.43	2.87	24.77	0.79
$210An_1$	108.27	1.10	46.33	0.32
$210An_2$	115.08	1.50	51.09	0.33

* 1st sampling after 0 week of incubation.

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE IV

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS AND METABOLITES FROM TREATMENT 3 CONTAINING SOIL TECHNICAL DDT, AND SULFUR, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	*p,p'*-DDD	*o,p'*-DDT	*p,p'*-DDE
$10A_1$***	10.04	1.73	4.76	1.12
$10A_2$	10.50	1.21	4.26	1.21
$110A_1$	56.91	7.66	24.35	0.89
$110A_2$	45.08	2.25	20.29	0.18
$210A_1$	94.31	13.47	41.19	0.21
$210A_2$	105.50	13.73	50.59	0.13
$10An_1$	5.71	2.56	2.58	0.93
$10An_2$	7.09	2.84	2.91	1.10
$110An_1$	28.58	5.72	9.53	0.78
$110An_2$	39.58	11.84	13.59	0.96
$210An_1$	80.02	19.61	32.77	0.21
$210An_2$	94.49	22.86	39.58	0.09

* 2nd sampling after 8 weeks of incubation

** As ppm oven-dry soil.

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 14

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT - COMPONENTS
AND METABOLITES FROM TREATMENT 3 CONTAINING
SOIL, TECHNICAL DDT AND SULFUR, INCUBATED
AT 37C AT FIELD CAPACITY AND BY FLOODING
WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	9.25	0.51	5.39	0.65
$10A_2$	9.47	0.20	3.31	1.10
$110A_1$	53.47	6.93	19.80	1.48
$110A_2$	69.78	3.67	26.63	1.84
$210A_1$	93.59	1.84	40.94	0.56
$210A_2$	104.83	10.39	47.65	1.47
$10An_1$	5.91	1.70	1.67	0.79
$10An_2$	5.67	2.52	2.25	0.81
$110An_1$	34.84	10.20	11.61	1.01
$110An_2$	50.50	10.89	16.33	1.48
$210An_1$	77.09	20.20	39.44	1.49
$210An_2$	78.84	19.21	27.94	1.22

* 3rd sampling after 12 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 11

GAS CHROMATOGRAPHIC ANALYSIS OF THE DDT - COMPONENTS
AND METABOLITES FROM TREATMENT J CONTAINING
SOIL, TECHNICAL DDT, AND SULFUR, INCUBATED
AT 37C AT FIELD CAPACITY AND BY FLOODING
WITH WATER *

Treatment number	DDT components and metabolites**			
	p p -DDT	p p -DDD	o p -DDT	p p -DDE
10A$_1$***	10.02	0.33	3.03	0.48
10A$_2$	6.45	0.12	2.54	0.67
110A$_1$	40.78	1.21	17.48	0.68
110A$_2$	46.83	1.80	15.74	0.64
210A$_1$	100.49	2.46	38.41	0.88
210A$_2$	112.20	3.60	47.47	1.00
10An$_1$	2.48	1.33	0.66	0.43
10An$_2$	3.63	1.04	0.68	0.39
110An$_1$	25.25	5.62	3.62	1.84
110An$_2$	34.00	7.73	7.73	0.43
210An$_1$	25.36	6.60	3.66	0.59
210An$_2$	52.60	13.15	12.62	0.53

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE [illegible]

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT-COMPONENTS AND METABOLITES FROM TREATMENTS CONTAINING SOIL, TECHNICAL DDT, AND SULFUR, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	9.29	0.04	2.70	0.57
$10A_2$	7.14	0.04	1.88	0.87
$110A_1$	48.55	4.80	15.42	0.34
$110A_2$	56.17	4.14	25.78	0.31
$210A_1$	96.16	7.48	43.93	1.96
$210A_2$	118.77	8.22	52.07	1.82
$10An_1$	9.46	3.42	2.61	1.20
$10An_2$	10.86	5.12	2.57	1.70
$110An_1$	37.37	15.15	12.63	0.50
$110An_2$	53.91	24.79	20.20	1.08
$210An_1$	103.51	40.20	36.16	3.51
$210An_2$	95.47	40.20	35.17	3.01

* 8th sampling after 24 weeks of incubation

** As ppm oven-dry soil.

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE [illegible]

GAS CHROMATOGRAPHIC ANALYSES OF [illegible]
AND METABOLITES FROM TREA[illegible]
SOIL, TECHNICAL DDT, AND CALCIU[illegible] CARBONATE
INCUBATED AT 37C AT FIELD CAPACITY AND BY
FLOODING WITH WATER *

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	6.85	1.31	2.78	1.08
$10A_2$	13.73	1.31	4.57	1.23
$110A_1$	48.50	7.57	22.73	3.03
$110A_2$	47.97	6.58	20.20	2.53
$210A_1$	136.25	7.73	61.84	2.89
$210A_2$	105.07	7.31	50.25	3.65
$10An_1$	7.30	0.59	2.65	1.84
$10An_2$	6.27	0.47	2.59	1.69
$110An_1$	75.27	4.76	25.73	0.65
$110An_2$	60.01	3.03	26.26	0.84
$210An_1$	118.95	1.70	51.27	0.41
$210An_2$	91.19	2.23	36.36	1.10

* 1st sampling after 0 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 22

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT-COMPONENTS AND METABOLITES FROM TREATMENT 4 CONTAINING SOIL TECHNICAL DDT AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	*p,p*'-DDD	*o,p*'-DDT	*p,p*'-DDE
$10A_1$***	3.35	1.26	3.70	1.10
$10A_2$	10.67	1.28	4.65	1.38
$110A_1$	50.49	1.33	23.03	0.88
$110A_2$	77.07	3.10	32.78	0.20
$210A_1$	102.45	13.87	52.00	0.82
$210A_2$	124.27	18.38	64.71	2.57
$10An_1$	6.43	2.48	2.16	0.96
$10An_2$	5.40	4.45	2.52	0.91
$110An_1$	38.36	9.22	12.62	0.24
$110An_2$	31.78	8.70	10.88	0.17
$210An_1$	83.83	27.24	35.63	0.96
$210An_2$	110.22	33.37	48.12	1.24

* 2nd sampling after 6 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE [illegible]

GAS CHROMATOGRAPHIC ANALYSES OF THE DDT- COMPONENTS
AND METABOLITES FROM TREATMENTS RECEIVING
SOIL, TECHNICAL DDT, AND CALCIUM CARBONATE,
INCUBATED AT 37C AT FIELD CAPACITY AND BY
FLOODING WITH WATER*

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	p,p'-DDD	o,p'-DDT	p,p'-DDE
$10A_1$***	8.37	1.28	3.31	1.28
$10A_2$	6.31	0.54	2.80	1.26
$110A_1$	53.38	5.40	22.86	2.38
$110A_2$	57.71	6.12	28.28	3.08
$210A_1$	105.88	5.48	45.68	2.30
$210A_2$	109.83	10.08	50.25	2.74
$10An_1$	4.50	2.16	1.38	0.95
$10An_2$	4.67	2.07	1.88	1.29
$110An_1$	25.02	11.06	7.22	1.44
$110An_2$	40.02	12.86	18.67	1.80
$210An_1$	59.27	21.38	18.82	1.46
$210An_2$	56.38	21.33	22.10	2.09

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* 3rd sampling after 12 weeks of incubation.

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 24

[illegible] ANALYSES OF THE [illegible]-COMPONENTS AND METABOLITES FROM TREATMENTS CONTAINING SOIL, TECHNICAL DDT, AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	DDT-components and metabolites**			
	p p -DDT	p p -DDD	o p -DDT	p p'-DDE
$10A_1$***	5.47	0.33	1.56	0.73
$10A_2$	12.58	0.14	3.38	0.88
$110A_1$	47.03	2.47	16.83	1.18
$110A_2$	40.35	2.29	16.52	1.10
$210A_1$	107.19	1.40	45.00	1.12
$210A_2$	101.00	1.26	47.47	2.72
$10An_1$	6.35	1.76	1.41	0.44
$10An_2$	3.02	1.47	0.33	0.28
$110An_1$	35.35	12.39	11.01	0.73
$110An_2$	27.88	3.46	8.94	0.73
$210An_1$	59.83	20.85	16.46	0.87
$210An_2$	60.49	17.68	16.83	0.74

---

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil.

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE [illegible]

[illegible]C ANALYSES OF [illegible] DDT - COMPONENTS AND METABOLITES FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT, AND CALCIUM CARBONATE INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	DDT-components and metabolites**			
	p,p'-DDT	*p,p'*-DDD	*o,p'*-DDT	*p,p'*-DDE
$10A_1$***	7.33	0.03	2.30	0.88
$10A_2$	10.24	0.26	2.38	0.79
$110A_1$	48.55	3.39	22.34	1.94
$110A_2$	50.50	3.03	22.22	1.51
$210A_1$	88.55	7.58	43.81	5.68
$210A_2$	114.02	8.68	52.18	6.76
$10An_1$	6.05	2.44	1.32	0.71
$10An_2$	6.27	3.09	1.32	0.87
$110An_1$	70.34	25.96	21.77	1.34
$110An_2$	41.71	14.22	14.22	0.07
$210An_1$	81.65	50.25	27.22	4.87
$210An_2$	106.15	64.21	33.48	5.58

---

* 5th sampling after 24 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

SUMMARY AND CONCLUSIONS

DDT has been extensively used during the past 20 years in controlling various insect pests. The extreme recalcitrance of DDT is well-documented (Edwards 1966). Persistence is an important characteristic contributing to the usefulness of DDT as an insecticide. Yet, this property contributes significantly to the harmful side effect of DDT application as a contaminant of man's foodchain and raises important questions concerning the movement and storage of this insecticide residue in soil.

The mechanisms of disappearance of DDT from soil are still poorly understood. According to the general trend in this field of pesticide research, it will be noted that interaction between pesticide chemicals and soil has been studied mostly under upland and not water-logged conditions. In general, a drastic effect is induced on biochemical and chemical activity in the soil by submergence.

The objective of this laboratory study was to investigate the fate of applied technical-grade DDT in virgin Arredondo fine sand and this soil which had been altered with additions of activated sewage, sulfur, and calcium carbonate and incubated at 37C under well-aerated and water-logged conditions.

The composition of the crude DDT varies with the preparation process employed and the conditions of reaction. It has been reported to be composed of at least 14 compounds, the principal ones being *p,p'*-DDT (65-80%), *o,p'*-DDT (11-30%), and *p,p'*-DDD (0.1-4%). *p,p'*-DDT is generally agreed to be the most active principle.

DDT persists in soil strongly resists microbial degradation, although dechlorination of DDT to DDD can occur in higher organisms (Datta et al. 1964). Also, dechlorination of DDT by microorganisms is possible (Barker and Morrison 1966, Kallman and Andrews 1963). However, the efficiency of conversion by microorganisms is inversely proportional to the availability of oxygen in the system (Stenersen 1965, Castro 1964, Miskus et al. 1965). It seems likely that microbial dechlorination of DDT might be explained as a process implicating the cytochromes of the respiratory chain, the absence of oxygen serving to keep the cytochromes in the reduced (Fe II) state. The use of selective inhibitors indicated that the reduced Fe (II) cytochrome oxidase was responsible for DDT dechlorination (Wedemeyer 1966). DDD is often detected as a residue in situations where only DDT has been used, and DDD appears to persist for unusually long periods. Studies with ^{14}C-DDT incubated with bovine rumen fluid, lake water, and aqueous solutions of reduced porphyrins showed partial conversion to ^{14}C-DDD (Miskus et al. 1965). ^{14}C-DDT and carrier DDT were added to soil and incubated under anaerobic condition at an atmosphere of 20% CO_2 and 80% N_2. DDT was dechlorinated to DDD in the anaerobic system by soil microorganisms (Guenzi and Beard 1967a).

Soil samples (0-15 cm) representing Arredondo fine sand were obtained from the University of Florida Campus. The activated sewage was obtained from the Sewage Treatment Plant also on the University of Florida Campus. All samples were characterized prior to the experimentation.

The factorial experiment was conducted in the completely randomized design. Technical-grade DDT powder, flowers of sulfur, and calcium carbonate were applied and thoroughly mixed with the soil. The purpose of sulfur and calcium carbonate treatments was to get extreme pH values [illegible] 10 ppm [illegible] ppm [illegible] at field capacity [illegible] were obtained by [illegible] of 2 inches [illegible] water over the soil [illegible] experiment was conducted [illegible] incubators at 37C. Soil samples were taken after 0, 6, 12, 18, [illegible] weeks of incubation and analyzed for DDT residues and metabolites. The soil pH was also determined.

The gas chromatography equipment used consisted of a number of separate units designed to fit together to form a complete chromatography system. The chromatograph was equipped with an electron-capture detector containing 200 mCi tritium foil. Technical-grade DDT used in the experiment was analyzed for p,p'-DDT, o,p'-DDT, and p,p'-DDD. The operating parameters of the gas chromatograph were adjusted for the detection and quantification of p,p'-DDT, o,p'-DDT, p,p'-DDD, and p,p'-DDE as the technical-grade DDT components and metabolites.

The four treatment combinations did not have any appreciable effect on the degradation of the insecticide. It was, therefore, decided to consider the average of p,p'-DDT, p,p'-DDD, o,p'-DDT, and p,p'-DDE separately from the four treatment combinations.

p,p'-DDT was dechlorinated to p,p'-DDD in the flooded soil. Analysis of the soil samples at the end of 0, 6, 12, 18 and 24 weeks of incubation showed that in the flooded soil treated with 10 ppm of technical-grade DDT, p,p'-DDT content decreased from around 8 ppm to 2.6 ppm, whereas, p,p'-DDD concentration rose from around 0.6 ppm to 2.8 ppm. The level of p,p'-DDD declined from 1 ppm to 0.14 ppm in the moist soil.

Because the degree of aeration is inversely related to soil moisture [illegible] by flooding with water oxygen will [illegible] a [illegible]). The observed dechlorination of p,p'-DDT and its conversion to p,p'-DDD was enhanced under water-logged conditions creating or favoring anaerobiosis.

In 110 ppm technical-grade DDT treatments, anaerobic conditions obtained by flooding caused reductive dechlorination of p,p'-DDT to the extent that a final concentration of 15.4 ppm of p,p'-DDD was recorded. The concentration of p,p'-DDT decreased from around 52 ppm to 33 ppm in the flooded soil only and no noticeable change occurred in the well-drained situation.

In 210 ppm technical-grade DDT treatments, at the end of incubation, 35 ppm of p,p'-DDD was detected in the soil kept under water. Analysis of the moist soil showed an increase to 10 ppm of p,p'-DDD presumably due to localized anaerobiosis caused by entrapped CO_2 or water. However, with time, p,p'-DDD degraded and a lower concentration of 3 ppm was detected. p,p'-DDT showed recalcitrance in the moist soil but in the flooded soil it decreased from 98 ppm to 60 ppm.

o,p'-DDT is the major constituent of technical-grade DDT (27.5%) and as a biocide is much less toxic than p,p'-DDT.

In 10 ppm technical-grade DDT treatments, o,p'-DDT breakdown occurred in the soil maintained at field capacity and in the water-logged state. The loss was more significant from the flooded soil where from a 3 ppm concentration at 0 time, 0.5 ppm was reached after 24 weeks of incubation.

In 110 ppm technical-grade DDT treatments, from a concentration around 23 ppm, o,p'-DDT degraded to 9 ppm under anaerobic conditions. The compound practically remained unaltered in the aerobic soil.

Some alteration of o,p'-DDT was noticeable in the moist soil containing 210 [illegible] initial concentration of 63 ppm at [illegible] *o,p*'-DDT was lost from the flooded soil where from an initial concentration of 40 ppm at the start of incubation only 17 ppm was detected at the end of 24 weeks

This study found similar amounts of *p,p*'-DDE in all of the three levels of technical-grade DDT treatments The concentrations ranging from 0.4 ppm to 2.6 ppm were not significantly different under both aerobic and anaerobic conditions.

Since temperature was not a variable in this investigation it may be safe to say that other than the indirect effect on soil microbes, it was not directly involved in the degradation of the insecticide.

The following conclusions were made

1 The four treatments consisting of sewage, no treatment, sulfur and calcium carbonate did not have any significant interaction with the levels of *p,p*'-DDT, *p,p*'-DDD, *o,p*'-DDT, and *p,p*'-DDE as degradation products of technical-grade DDT It is also apparent that pH changes did not affect the breakdown of DDT.

2 Anaerobic conditions obtained by flooding caused reductive dechlorination of *p,p*'-DDT and its conversion to *p,p*'-DDD was enhanced under water-logged conditions creating or favoring anaerobiosis

3. Aerobic conditions favored the degradation of *p,p*'-DDD An increase in the concentration of *p,p*'-DDD at the beginning in the moist soil was presumably due to localized anaerobiosis caused by entrapped CO_2 or water.

4. In general, more *o,p*'-DDT was lost from the flooded soil and some alteration of this compound was noticeable in the moist soil.

[illegible] amounts of [illegible] at the three levels of technical-grade [illegible] treatments and the concentrations were not significantly different under both aerobic and anaerobic conditions

APPENDIX

TABLE 28

ANALYSIS OF VARIANCE FOR pH CHANGES IN FOUR DIFFERENT COMBINATIONS WITH THREE LEVELS OF TECHNICAL DDT, INCUBATED AT [illegible] UNDER AEROBIC AND ANAEROBIC CONDITIONS

Source	Degrees of freedom	Mean squares				
		Weeks of incubation				
		0	6	12	16	[illegible]
Treatment combinations (T)	3	1.3301**	15.8060**	23.2171**	21.9559**	26.4[illegible]
DDT levels (L)						
Linear	1	0.0045	0.0095	0.0226	0.0028	[illegible]
Quadratic	1	0.0040	0.1169**	0.1709**	0.0417*	[illegible]
T x L	6	0.0119	0.1269**	0.0571**	0.0922**	[illegible]
Field capacity versus flooding (A)	1	0.0016	6.1994**	1.9805**	2.0213**	1.5643
T x A	3	0.0003	0.9017**	0.0289**	0.0806**	[illegible]
L x A	2	0.0028	0.0480*	0.0170	0.0163	[illegible]
T x L x A	6	0.0080	0.0284	0.0418**	0.0117	[illegible]
Error	24	0.0084	0.0123	0.0054	[illegible]	[illegible]

* Significant at the 5% level

**Significant at the 1% level

TABLE 27

ANALYSIS OF VARIANCE FOR DDT - COMPONENTS AND METABOLITES IN FOUR TREATMENT COMBINATIONS WITH THREE LEVELS OF TECHNICAL DDT, INCUBATED AT 37C UNDER AEROBIC AND ANAEROBIC CONDITI

Source	Degrees of freedom	Mean square Weeks of incubation 0	 6	 12	 18	 24
Treatment combinations (T)	3	132.73**	83.81*	46.40*	198.30**	221.[illegible]
DDT levels (L)						
Linear	1	42587.48**	40917.31**	28205.90**	26677.90**	[illegible]
Quadratic	1	35.56	37.28	0.14	204.18**	[illegible]
T x L	6	146.64**	184.60**	50.61**	68.02**	[illegible]
Field capacity versus flooding (A)	1	267.79**	644.34**	1748.22**	1680.76**	486.[illegible]
T x A	3	33.94	30.78	63.02**	144.35**	948.71**
L x A	2	739.75**	153.76**	345.98**	836.23**	104.25
T x L x A	6	14.32	88.63**	57.90**	50.62**	172.87*
DDT - components and metabolites (C)	3	31838.84**	24312.66**	18474.37**	16003.45**	23682.18**
T x C	9	35.70	24.38	30.02*	37.77*	39.14
L x C	6	8430.16**	6367.52**	4206.52**	4689.60**	5278.10**
T x L x C	18	67.20*	23.38	17.22	13.88	60.31
A x C	3	20.90	891.21**	1779.65**	1886.53**	2339.38**
T x A x C	9	26.87	19.30	33.85*	24.55	167.05*
L x A x C	6	234.08**	172.35**	366.84**	637.10**	566.06**
T x L x A x C	18	9.17	35.94	26.23*	15.19	71.23
Error	96	32.49	21.85	13.81	14.[illegible]	63.71

* Significant at the 5% level
**Significant at the 1% level

TABLE 28

pH CHANGES AND COLORIMETRIC ANALYSES OF DDT FROM TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.30	11.28
$10A_2$	6.25	10.70
$110A_1$	6.30	101.03
$110A_2$	6.35	102.79
$210A_1$	6.30	163.69
$210A_2$	6.20	184.46
$10An_1$	6.35	10.21
$10An_2$	6.20	9.71
$110An_1$	6.25	91.65
$110An_2$	6.40	82.85
$210An_1$	6.35	160.52
$210An_2$	6.30	173.12

* 1st sampling after 0 week of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

[illegible]

pH CHANG[illegible] DDT TREATMEN[illegible] TECHNICAL DDT AND ACTIVATED [illegible] [illegible]ED AT 37C AT FIELD CAPACI[illegible] [illegible] FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.10	9.97
$10A_2$	6.00	9.26
$110A_1$	5.90	93.84
$110A_2$	5.90	83.47
$210A_1$	5.90	169.53
$210A_2$	5.90	184.95
$10An_1$	6.45	10.26
$10An_2$	6.35	9.83
$110An_1$	6.40	86.33
$110An_2$	6.30	100.27
$210An_1$	6.10	190.94
$210An_2$	6.10	178.41

* 2nd sampling after 6 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

[illegible]

[illegible] AND COLORIMETRIC ANALY[illegible] TREATMENT 1 CONTAINING SOIL, TECHN[illegible] ACTIVATED SEWAGE INCUBATED AT 37C AT [illegible] CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.80	10.71
$10A_2$	5.75	10.23
$110A_1$	5.60	97.81
$110A_2$	5.50	88.09
$210A_1$	5.50	200.55
$210A_2$	5.45	202.69
$10An_1$	6.20	9.48
$10An_2$	6.00	10.08
$110An_1$	6.00	106.30
$110An_2$	6.10	98.16
$210An_1$	5.80	176.32
$210An_2$	6.00	168.48

* 3rd sampling after 12 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 21

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT OF TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT AND ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.20	10.66
$10A_2$	5.70	10.12
$110A_1$	5.65	85.92
$110A_2$	5.50	84.42
$210A_1$	5.60	175.26
$210A_2$	5.50	182.85
$10An_1$	6.35	8.70
$10An_2$	6.40	8.54
$110An_1$	6.05	100.64
$110An_2$	6.15	97.44
$210An_1$	5.80	184.34
$210An_2$	6.00	170.45

* 4th sampling after 12 weeks of incubation

** As ppm oven-dry soil.

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 32

pH CHANGES AND COLORIMETRIC ANALYSES [illegible] TREATMENT 1 CONTAINING SOIL, TECHNICAL DDT [illegible] ACTIVATED SEWAGE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.40	11.13
$10A_2$	5.35	11.02
$110A_1$	5.30	89.44
$110A_2$	5.35	101.35
$210A_1$	5.20	146.22
$210A_2$	5.25	128.85
$10An_1$	5.80	9.95
$10An_2$	6.05	9.56
$110An_1$	5.80	96.86
$110An_2$	5.85	84.73
$210An_1$	5.75	185.00
$210An_2$	5.80	184.02

* 5th sampling after 24 weeks of incubation.

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 33

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.30	10.01
$10A_2$	6.20	10.47
$110A_1$	6.30	87.19
$110A_2$	6.30	106.28
$210A_1$	6.30	177.36
$210A_2$	6.25	184.53
$10An_1$	6.35	10.34
$10An_2$	6.25	9.51
$110An_1$	6.25	88.04
$110An_2$	6.35	85.66
$210An_1$	6.30	181.81
$210An_2$	6.35	189.66

* 1st sampling after 0 week of incubation

** As ppm oven-dry soil.

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE [illegible]

pH [illegible] ANALYSES FOR DDT FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.70	9.75
$10A_2$	5.90	9.05
$110A_1$	5.80	98.12
$110A_2$	5.90	97.19
$210A_1$	5.85	203.32
$210A_2$	5.65	173.49
$10An_1$	6.45	10.08
$10An_2$	6.35	11.19
$110An_1$	6.40	92.92
$110An_2$	6.60	103.66
$210An_1$	6.20	185.40
$210An_2$	6.40	193.89

* 2nd sampling after 8 weeks of incubation.

** As ppm oven-dry soil.

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 35

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.50	9.72
$10A_2$	5.45	10.43
$110A_1$	5.70	93.52
$110A_2$	5.65	84.24
$210A_1$	5.45	162.37
$210A_2$	5.35	178.83
$10An_1$	5.90	9.34
$10An_2$	5.85	3.82
$110An_1$	6.00	90.25
$110An_2$	6.05	90.75
$210An_1$	5.80	172.20
$210An_2$	5.80	168.97

* 3rd sampling after 12 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 38

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 2 CONTAINING SOIL AND TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.60	8.59
$10A_2$	5.40	10.38
$110A_1$	5.40	97.33
$110A_2$	5.30	102.78
$210A_1$	5.45	201.03
$210A_2$	5.35	172.53
$10An_1$	6.00	8.50
$10An_2$	6.05	9.08
$110An_1$	6.05	88.63
$110An_2$	6.30	91.88
$210An_1$	5.85	198.55
$210An_2$	5.95	174.44

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE [illegible]

[illegible] FOR DDT FROM [illegible] CONTAINING [illegible] TECHNICAL DDT, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.60	10.80
$10A_2$	5.65	10.40
$110A_1$	5.60	89.73
$110A_2$	5.50	98.58
$210A_1$	5.40	171.48
$210A_2$	5.38	154.79
$10An_1$	6.10	8.54
$10An_2$	6.05	7.89
$110An_1$	5.98	83.19
$110An_2$	6.01	81.53
$210An_1$	5.78	164.79
$210An_2$	5.70	184.61

* 5th sampling after 24 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 38

[illegible] ETRIC ANALYSES FOR DDT FROM
[illegible] CONTAINING SOIL, TECHNICAL DDT, AND
[illegible] AT 37C AT FIELD CAPACITY AND
[illegible] FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.55	10.72
$10A_2$	6.20	9.80
$110A_1$	6.05	98.61
$110A_2$	6.15	90.32
$210A_1$	6.25	191.89
$210A_2$	6.15	185.07
$10An_1$	6.35	10.69
$10An_2$	6.20	9.87
$110An_1$	6.30	96.31
$110An_2$	6.30	99.53
$210An_1$	6.15	180.81
$210An_2$	6.20	193.56

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* 1st sampling after 0 week of incubation

** As ppm oven-dry soil.

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

[illegible]

pH C[illegible] TREAT[illegible] AND SULFUR INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

[illegible] number	pH(H_2O)	DDT**
10A$_1$***	3.45	10.02
10A$_2$	3.30	8.92
110A$_1$	3.40	96.49
110A$_2$	3.60	95.49
210A$_1$	3.35	180.81
210A$_2$	3.35	194.28
10An$_1$	5.00	9.43
10An$_2$	5.00	9.43
110An$_1$	5.00	90.01
110An$_2$	4.80	94.80
210An$_1$	4.95	198.95
210An$_2$	4.85	181.88

* 2nd sampling after 6 weeks of incubation

** As ppm oven-dry soil.

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 40

pH CHANGES AND COLORIMETRIC ANALYSIS FOR DDT FROM TREATMENTS CONTAINING SOIL, TECHNICAL DDT, AND SULFUR INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	3.20	9.96
$10A_2$	3.20	9.70
$110A_1$	3.30	94.87
$110A_2$	3.35	90.85
$210A_1$	3.30	182.85
$210A_2$	3.25	164.34
$10An_1$	3.50	10.04
$10An_2$	3.40	10.06
$110An_1$	3.60	97.93
$110An_2$	3.50	87.48
$210An_1$	3.60	175.25
$210An_2$	3.65	189.00

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* 3rd sampling after 12 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 41

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 3 CONTAINING SOIL, TECHNICAL DDT AND SULFUR, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	3.35	10.19
$10A_2$	3.35	10.41
$110A_1$	3.40	88.40
$110A_2$	3.35	100.18
$310A_1$	3.35	182.65
$210A_2$	3.35	176.84
$10An_1$	3.65	10.28
$10An_2$	3.55	8.72
$110An_1$	3.50	98.32
$110An_2$	3.55	87.25
$210An_1$	3.50	155.10
$310An_2$	3.60	168.68

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

TABLE 42

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT [illegible] TREATMENT 3 CONTAINING SOIL, TECHNICAL DDT AND SULFUR, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	3.05	10.41
$10A_2$	3.00	9.15
$110A_1$	3.10	103.65
$110A_2$	3.00	84.36
$210A_1$	2.95	175.35
$210A_2$	3.00	161.32
$10An_1$	3.75	10.07
$10An_2$	3.45	9.58
$110An_1$	3.80	89.28
$110An_2$	3.15	97.58
$210An_1$	2.80	149.54
$210An_2$	3.10	157.08

* 5th sampling after 24 weeks of incubation.

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 41

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 4 CONTAINING SOIL, TECHNICAL DDT AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER[*]

Treatment number	pH(H_2O)	DDT[**]
$10A_1$[***]	6.85	10.09
$10A_2$	7.00	9.02
$110A_1$	6.95	104.08
$110A_2$	7.00	87.29
$210A_1$	6.95	185.85
$210A_2$	6.85	178.86
$10An_1$	6.82	5.66
$10An_2$	7.00	9.18
$110An_1$	6.80	90.90
$110An_2$	7.05	78.34
$210An_1$	6.95	180.90
$210An_2$	6.85	178.92

[*] 1st sampling after 0 week of incubation.

[**] As ppm oven-dry soil.

[***] Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE 11

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 4 CONTAINING SOIL, TECHNICAL DDT AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.00	10.23
$10A_2$	6.30	9.14
$110A_1$	6.75	100.84
$110A_2$	6.85	92.86
$210A_1$	6.95	184.73
$210A_2$	7.05	178.79
$10An_1$	7.00	9.82
$10An_2$	6.80	10.39
$110An_1$	7.10	96.33
$110An_2$	7.30	88.09
$210An_1$	7.20	193.49
$210An_2$	7.00	178.61

* 2nd sampling after 6 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times

[illegible]

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 4 CONTAINING SOIL, TECHNICAL DDT, AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	5.80	10.42
$10A_2$	6.00	10.03
$110A_1$	6.60	61.81
$110A_2$	6.70	84.46
$210A_1$	6.65	172.15
$210A_2$	6.60	169.33
$10An_1$	6.70	10.08
$10An_2$	6.60	8.13
$110An_1$	6.65	86.45
$110An_2$	7.10	87.60
$210An_1$	6.90	173.15
$210An_2$	6.80	163.60

* 3rd sampling after 12 weeks of incubation

** As ppm oven-dry soil

***Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

[illegible]

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 4 CONTAINING SOIL TECHNICAL DDT, AND CALCIUM CARBONATE, INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER *

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.10	9.87
$10A_2$	6.25	10.03
$110A_1$	6.45	82.64
$110A_2$	6.65	81.81
$210A_1$	6.50	140.88
$210A_2$	6.70	148.76
$10An_1$	6.50	10.23
$10An_2$	6.55	9.74
$110An_1$	6.85	91.82
$110An_2$	6.80	83.06
$210An_1$	6.85	141.06
$210An_2$	6.85	156.13

* 4th sampling after 18 weeks of incubation

** As ppm oven-dry soil

*** Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

TABLE [illegible]

pH CHANGES AND COLORIMETRIC ANALYSES FOR DDT FROM TREATMENT 4 CONTAINING SOIL, TECHNICAL DDT AND CALCIUM CARBONATE INCUBATED AT 37C AT FIELD CAPACITY AND BY FLOODING WITH WATER.*

Treatment number	pH(H_2O)	DDT**
$10A_1$***	6.15	10.77
$10A_2$	6.25	9.91
$110A_1$	6.55	78.68
$110A_2$	6.80	83.62
$210A_1$	6.55	133.68
$210A_2$	6.70	157.58
$10An_1$	6.60	10.45
$10An_2$	6.65	10.69
$110An_1$	6.90	93.46
$110An_2$	6.85	85.48
$210An_1$	6.95	150.26
$210An_2$	6.85	157.75

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[*] 5th sampling after 24 weeks of incubation.

[**] As ppm oven-dry soil.

[***] Technical-grade DDT powder in ppm applied in the soil and replicated 2 times.

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BIOGRAPHICAL SKETCH

[illegible] Bhuiya was born January 1, 1932, at Village Naoghat, Brahmanbaria, E. Pakistan. He passed the Matriculation Examination in 1948. In 1952, he received the degree of Bachelor of Science in first division with distinction from the University of Dacca, and stood third in order of merit in the province of E. Pakistan. He received the Master of Science degree in Soil Science, Part I, in 1953, and Part II in 1954, standing first in the first class from the same University.

He entered the government service in November, 1954, in the Irrigation Department and served as Research Assistant. In December 1955, he joined the University of Dacca as Junior Lecturer and taught Soil Science and Geology. He was promoted to Lecturer in 1957. He was awarded the Colombo Plan Scholarship from the Government of Canada and obtained the Master of Science in Agriculture degree from the Ontario Agricultural College and the University of Toronto in 1961. He had an opportunity to become familiar with the organization and operation of soil classification and land use planning work in Ontario. He visited several research stations in Ontario to study research programs underway.

He joined the E. Pakistan Agricultural University in July, 1963, as Senior Lecturer. From September 30 to November 25, 1963, he participated and completed the course of study on the application of radioactive isotopes in soil-plant relations, carried out jointly by the International Atomic Energy Agency and the Government of Turkey, held at the Isotope Laboratory of the Faculty of Agriculture, University of Ankara.

[illegible] Soil Science and was on [illegible] studies in Soil Microbiology. In September, 1965, he joined the University of Florida to work towards the degree of Doctor of Philosophy in the Soils Department, College of Agriculture, with Soil Chemistry and Bacteriology as the major and minor subjects, respectively.

He is a council member of the Soil Science Society of Pakistan. He has published one book and several articles in national and international journals.

He is married, and the father of four children.

This dissertation was prepared under the direction of the chairman of the candidate's supervisory committee and has been approved by all members of that committee. It was submitted to the Dean of the College of Agriculture and to the Graduate Council, and was approved as partial fulfillment of the requirements for the degree of Doctor of Philosophy.

June 1968

Dean, College of Agriculture

Dean, Graduate School

Supervisory Committee:

Chairman